

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

NATIONAL EXPOSURE RESEARCH LABORATORY RESEARCH TRIANGLE PARK, NC 27711

OFFICE OF RESEARCH AND DEVELOPMENT

February 4, 2019

Ken Kloo, Director NJ Department of Environmental Protection Division of Remediation Management Mail Code 401-05M 401 East State Street P.O. Box 420 Trenton, NJ 08625-0420

Subject: NJ DEP Report #1: Targeted Analysis of PFCA in Soil Samples

Dear Mr. Kloo:

I am pleased to provide you with this initial laboratory report of targeted analysis results for perfluorinated carboxylic acid (PFCA) concentrations in soils. This is the first in a series of reports prepared as a part of EPA Office of Research and Development's (ORD) collaboration with the New Jersey Department of Environmental Protection (NJ DEP) and EPA Region 2 on the study, "Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Substances (PFAS) in Environmental Media from an Industrialized Area of New Jersey." This report includes concentration results for 13 PFCA in 24 soil samples. The ORD Principal Investigators (PIs) for this study are Drs. Andy Lindstrom, Mark Strynar, and John Washington. The results for this particular report were generated by Dr. John Washington in our Athens, Georgia laboratory. It is my understanding that these samples were collected by NJ DEP between October 23, 2017 and November 11, 2017 from various locations in the vicinity of the Solvay and Dupont facilities.

We do not interpret exposure or risk from the values presented in this report. EPA does not currently have health-based standards, toxicity factors, or associated risk levels for per- or polyfluorinated alkyl substances (PFAS), other than perfluorocatanoic acid (PFOA) and perfluorocatane sulfonate (PFOS). While the data provided indicate the presence of certain PFCA in soil samples, it does not offer interpretation as to human or environmental exposure or risk.

Thank you for providing us with this opportunity for collaboration that helps to further both EPA's and New Jersey's understanding of an important public health issue. If you have any questions or concerns about this report, do not hesitate to contact me at (919) 541-2107 or via email at watkins.tim@epa.gov or Tim Buckley at (919) 541-2454 or via email at Buckley.timothy@epa.gov. I look forward to our continued work together.

Sincerely,

Timothy H. Watkins

Director

National Exposure Research Laboratory Office of Research and Development

Enclosure

CC:

Nidal Azzam, USEPA, Region 2 Daniel D'Agostino, USEPA, Region 2 Jeff Morris, USEPA OPPT Betsy Behl, USEPA, OW Andy Gillespie, USEPA, ORD Timothy Buckley, USEPA, ORD Mark Strynar, USEPA, ORD Andy Lindstrom, USEPA, ORD John Washington, USEPA, ORD NJ DEP Report #1 February 4, 2019

Detection, Evaluation, and Assignment of PFAS in Environmental Media from an Industrialized Area of New Jersey

Laboratory Data Report #1: Targeted Analysis of PFCA in Soil

Background. EPA/ORD, EPA Region 2, and New Jersey Department of Environmental Protection (NJ DEP) worked together to develop a study to evaluate sources, as well as the nature and extent of PFAS contamination near manufacturing facilities in NJ. NJ DEP assumed responsibility for the collection of samples and their shipment to ORD laboratories. ORD was responsible for sample extraction and analysis. ORD personnel involved with laboratory analysis and their roles and responsibilities are provided below in Table 1.

| Table 1. EPA | . Office of Research | and Development | Analysis and Re | port Team. |
|--------------|----------------------|-----------------|-----------------|------------|
| | | | | |

| Responsibility | Personnel |
|------------------------------------|--------------------------------------|
| ORD Principal Investigators | Andy Lindstrom, Mark Strynar, and |
| | John Washington |
| Laboratory Chemistry | John Washington (PI) and Tom Jenkins |
| Quality Assurance Review | Brittany Stuart, Sania Tong-Argao |
| Management Coordination and Review | Brian Schumacher, Adam Biales, |
| | Myriam Medina-Vera, Tim Buckley |
| Report Preparation | Kate Sullivan, Tim Buckley |

This report includes results for 24 soil samples and 4 duplicates and field blanks collected by NJ DEP from November 8-10, 2017 and delivered to the ORD lab in Athens, GA on November 14, 2017. The results provided in this report were analyzed under the direction of Dr. John Washington. This report does not contain results for 5 soil core samples collected at the same time as analysis is not complete.

Thirteen perfluorinated carboxylic acid (PFCA) listed in Table 2 were analyzed with Ultra-Performance Liquid Chromatography Mass Spectrometry (UPLC-MS) using methods described within our Quality Assurance Project Plan (QAPP)¹ and that have been generally described in Rankin et al., 2015². These analytes were selected because previous reports have shown them to be of concern. In brief, each sample was divided into three ~1 g aliquots. Each aliquot was extracted with 90%/10% acetonitrile water followed by a liquid/liquid cleanup. Samples were analyzed using a Waters Acquity UPLC coupled to a Waters Quattro Premier XE tandem mass spectrometer. The reported concentrations are determined as the mean value of triplicate aliquot analysis (Table 3). PFCA concentrations were determined using mass-labeled internal calibration curves for quantitation using a traditional targeted analysis approach. These analyses were performed on samples, process blanks, and check standards. Dilution of samples was not performed.

¹ National Exposure Research Laboratory, Quality Assurance Project Plan: Detection, Evaluation and Assignment of Multiple Poly and Per-fluoroalkyl Substances (PFAS) in environmental media from an industrialized area of New Jersey. Prepared for New Jersey Department of Environmental Protection (NJ DEP), September 14, 2017.

² K. Rankin, S. A. Maybury, T.M. Jenkins, J.W. Washington, A North American and global survey of perfluoroalkyl substances in surface soils: Distribution patterns and mode of occurrence. Chemosphere 161, 333-341 (2015).

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Table 2. PFCA Analyzed in NJ Soil Samples by UPLC-MS.

| Acronym | Chemical Name | Formula | CAS no. | Monoisotopic Mass (g/mol) |
|---------|-----------------------------|---|------------|------------------------------|
| PFBA | Perfluorobutanoic Acid | C ₄ HF ₇ O ₂ | 375-22-4 | 213.9865 |
| PFPeA | Perfluoropentanoic Acid | $C_5HF_9O_2$ | 2706-90-3 | 263.9833 |
| PFHxA | Perfluorohexanoic Acid | $C_6HF_{11}O_2$ | 307-24-4 | 313.9801 |
| PFHpA | Perfluoroheptanoic Acid | C ₇ HF ₁₃ O ₂ | 375-85-9 | 363.9769 |
| PFOA | Perfluorooctanoic Acid | C ₈ HF ₁₅ O ₂ | 335-67-1 | 413.9737 |
| PFNA | Perfluorononanoic Acid | C ₉ HF ₁₇ O ₂ | 375-95-1 | 463.9705 |
| PFDA | Perfluorodecanoic Acid | C ₁₀ HF ₁₉ O ₂ | 335-76-2 | 513.9673 |
| PFUnDA | Perfluoroundecanoic Acid | C11HF21O2 | 2058-94-8 | 563.9641 |
| PRDoDA | Perfluorododecanoic Acid | C12HF23O2 | 307-55-1 | 613.9609 |
| PFTrDA | Perfluorotridecanoic Acid | C ₁₃ HF ₂₅ O ₂ | 72629-94-8 | 663.9577 |
| PFTeDA | Perfluorotetradecanoic Acid | C ₁₄ HF ₂₇ O ₂ | 376-06-7 | 713.9545 |
| PFHxDA | Perfluorohexadecanoic Acid | C ₁₆ HF ₃₁ O ₂ | 67905-19-5 | 813.9482 |
| PFODA | Perfluorooctadecanoic Acid | C ₁₈ HF ₃₅ O ₂ | 16517-11-6 | 913.9418 |

Quality Assurance

Data were checked for compliance with a number of laboratory and field related quality control evaluation criteria as specified in the project QAPP. Quality control results indicated that analyses were generally within expected performance criteria. Some individual analyte/sample values failed criteria and are flagged accordingly in Table 3.

Results

Concentration results for 24 soil samples identified by sample IDs assigned by NJ DEP are presented in Table 3 along with 2 field duplicates and 2 field blanks. Results are reported for 13 PFCAs that range from C4 through C18. Soil sample and duplicate concentrations are reported as the mean of three sample aliquots in units of mass of PFCA per unit mass of dry solid, i.e., pg/g.

- Concentrations across all analytes and samples ranged from <LOD to values that
 exceeded our calibration curve. In general, lower concentrations were observed for C4C8, C16, and C18 based on the number of values that were either <LOD or <LOQ.
 Across all samples, the highest concentrations were observed for C11 where two samples
 (PFSS001 and PFSS008) exceeded our calibration curve with estimated concentrations of
 5,730 and 6,560 pg/g, respectively. These values are flagged accordingly with "JC1".
- Mid-carbon length compounds (PFOA, PFNA, PFDA, and PFUA) were consistently present in higher concentrations than other compounds, although concentrations were variable from site to site. The odd-chain lengths C9, C11, and C13 exceeded the one-carbon homologues, C8, C10 and C12, in numerous samples, a pattern that is unusual in our experience.

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Table 3. PFCA Concentrations (pg/g) in Soil Samples Determined with Targeted Analysis.

| Carbon Length | 25 | S | છ | <i>C</i> 2 | 8 | 63 | C10 | C11 | C12 | C13 | C14 | 913 | C18 |
|---|----------------|-----------------|---|------------|----------|-----------|---------------|---|---------------|-----------------|------------|----------|-----------|
| NJDEP Sample ID | PFBA | PFPeA | PFHxA | РҒНрА | PFOA | PFNA | PFDA | PFUA | PFDoA | PFTrA | PFTeA | PFHxDA | PFODA |
| PFSS001 | 121 (U) | 143 | QN | 91.4 (U) | 398 | 2710 | 381 | 5730 (JC1) | 99.4 | 590 | 24.7 | ND | ND |
| PFSS002 | 102 (U) | 52.2 (U) | QN | 31.1 (U) | 196 (JP) | 91.2 | 115 | 1480 | 64.7 | 153 | 24.9 | QN | ND |
| PF5S003 | 149 | 153 | QN | 159 | 422 | 770 | 581 | 1440. | 244 | 252 | 104 | QN | GN |
| PFSS004 | ND | 366 (U) | 296 (U) | 345 | 1070 (U) | 2630 (JP) | 625 (U) | 2770 (JP) | 183 (U) | 326 (JP) | 81.6 (U) | QN | ND |
| PFSS005 | 112 (U) | 178 | ON | 132 | 244 (U) | 691 | 231 | 825 | 6.09 | 85.6 | 28.0 | QN | ND |
| PFSS006 | 119 (U) | 245 | QN | 102 | 295 (U) | 992 | 278 | 1370 | 111 | 165 | 46.5 | QN | ND |
| PFSS007 | 257 | 276 | 186 (U) | 188 | 749 | 1220 | 256 | 810 | 63.3 | 91.6 | 39.9 | GN | ND |
| PFSS008 | 69.4 (U) | 53.3 | QN | 37.7 (U) | QN | 295 | 324 | 6560 (JC1) | 277 | 1280 | 71.5 | QN | QN |
| PFSS009 | 103 | 104 | Q. | 91.2 | 162 (U) | 522 | 207 | 1460 | 84.7 | 226 | 35.4 | QN | ND |
| PFSS010 | QN | 77.7 | 158 (U) | 151 | 1900 | 286 | 262 | 794 | 100 | 104 | 39.8 | GN | ND |
| PFSS011 | 192 | 176 (U) | 155 (U) | 100 | 317 | 784 | 422 | 1370 | 117 | 166 | 54.9 | GN | QN |
| PFSS012 | 88.8 (U) | 171 | QN | 130 | 573 | 445 | 268 | 655 | 120 | 97.4 | 47.5 | GN | QN |
| PFSS013 | 159 | 199 | 426 (U) | 135 | 619 | 1150 | 312 | 1250 | 146 | 187 | 69.7 | QN | ND |
| PFSS014 | 228 (JP) | 192 | 228 (U) | 139 | 415 (JP) | (dr) 288 | 337 | 803 | 103 | 6.76 | 36.9 | QN | QN |
| PFSS015 | 153 (U) | 213 (JP) | 255 (U) | 163 (JP) | 599 (JP) | 614 (JP) | 473 (JP) | 3970 (JP) | 145 (JP) | 347 (JP) | 50.5 (JP) | S | ON |
| PFSS016 | 91.4 | 213 | 268 (U) | 172 | 264 | 651 | 296 | 307 | 74.1 | 54.3 | 31.5 | GN | ND |
| PFSS017 | 141 | 295 | 320 | 319 | 405 | 711 | 1460 | 1160 | 448 | 134 | 114 | 39.0 (U) | ND |
| PFSS018 | (U) 611 | 96.1 | Q | 66.3 | 160 (U) | 356 | 364 | 377 | 113 | 65.1 | 45.0 | GN | ND |
| PFSS019 | 143 (U) | 150 (U) | ND | (n) 8.39 | 151 (U) | 337 (U) | 193 | 543 | 83.8 | 78.7 | 38.0 | QN | ND |
| PFSS020 | 65.1 (U) | 121 | ND | 56.4 | 92.7 (U) | 475 | 192 | 1340 | 54.6 | 112 | 19.1 | GN | ND |
| PFSS021 | 133 (U) | 247 | Q | 176 | 689 | 626 | 401 | 895 | 150 | 129 | 71.2 | 8 | ND |
| PFSS022 | 092 | 622 | ND | 292 | 1250 | 658 | 269 | 1020 | 447 | 300 | 257 | 75.9 (U) | 86.5 (JP) |
| PFSS023 | 283 (JP) | 381 | 226 (U) | 446 | 330 (N) | 446 | 1060 | 1080 | 525 | 203 | 251 | 106 (U) | 30.8 (U) |
| PFSS024 | 257 | 425 | 493 (U) | 426 | 892 | 731 | 755 | 844 | 199 | 87.3 | 70.9 | ND | ND |
| PFSSDUP1 | 152 (U) | 535 | 526 (U) | 209 | 804 | 862 | 765 | 858 | 193 | 90.5 | 70.8 | QN | ON |
| PFSSDUP3 | 166 | 334 | ND | 94.2 | 185 (U) | 846 | 241 | 1080 | 84.9 | 122 | 42.0 | QN | ND |
| PFSSFB1 | 6.88 (U) | 6.96 (U) | ON | Q | ND | 7.94 | QN | ND | 1.81 | GN | 1.04 (U) | QN | QN |
| PFSSFB2 | ND | QN | ND | 7.52 (U) | QN | 10.1 | ND | 1.25 (U) | 13.7 | ON | 6.37 | QN | ND |
| ND: Sample result is less than the limit of detection (<lod).< th=""><th>ss than the li</th><th>mit of detectic</th><th>on (<lod).< th=""><th></th><th></th><th>; dí</th><th>ample triplic</th><th>Sample triplicates do not meet acceptance criteria for precision.</th><th>et acceptanc</th><th>ce criteria for</th><th>orecision.</th><th></th><th></th></lod).<></th></lod).<> | ss than the li | mit of detectic | on (<lod).< th=""><th></th><th></th><th>; dí</th><th>ample triplic</th><th>Sample triplicates do not meet acceptance criteria for precision.</th><th>et acceptanc</th><th>ce criteria for</th><th>orecision.</th><th></th><th></th></lod).<> | | | ; dí | ample triplic | Sample triplicates do not meet acceptance criteria for precision. | et acceptanc | ce criteria for | orecision. | | |
| U: Sample result is less than the limit of quantitation (<loq)< th=""><th>s than the lim</th><th>it of quantitat</th><th>ion (<loq).< th=""><th></th><th></th><th>)C1:</th><th>Sample resu</th><th>JC1: Sample result exceeds the upper calibration range</th><th>upper calibra</th><th>tion range.</th><th></th><th></th><th></th></loq).<></th></loq)<> | s than the lim | it of quantitat | ion (<loq).< th=""><th></th><th></th><th>)C1:</th><th>Sample resu</th><th>JC1: Sample result exceeds the upper calibration range</th><th>upper calibra</th><th>tion range.</th><th></th><th></th><th></th></loq).<> | | |)C1: | Sample resu | JC1: Sample result exceeds the upper calibration range | upper calibra | tion range. | | | |